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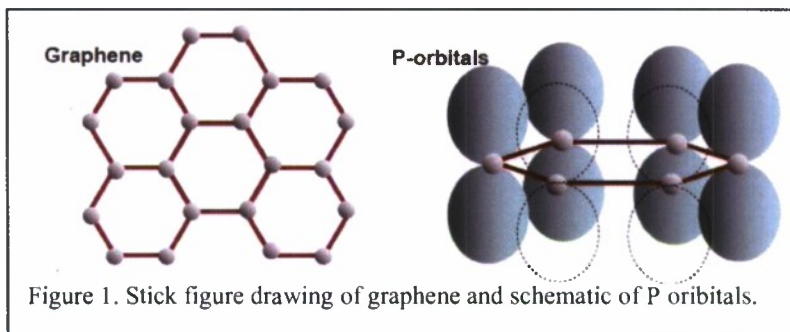
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14. ABSTRACT During the course of this work atmospheric and UHV techniques were developed for the production of few layer graphene on the Si and C face of SiC. It was found that control of the rate of Si loss (as determined by the surface temperature gradient) was necessary to produce high quality films on both the Si and C face. The quality of these films was confirmed by AFM, Hall, Raman, and high speed laser techniques. In particular the disorder ratio as determined by Raman analysis was less than .02. The results of the growth investigations were applied to the development of a commercial graphene materials growth reactor. One monolayer self supporting films of graphene were produced by a novel technique which under etched the SiC below the formed graphene. These self supporting layers have been used in fundamental studies on the mechanical and transport properties of graphene. Using the material developed in the project graphene gas sensors have been demonstrated. The sensors can operate in the amperometric mode or potentiometric mode. These sensors show excellent sensitivity for NO ₂ (less than 10ppb) but significantly					
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FINAL REPORT: GROWTH OF THIN HETERO-EPITAXIAL LAYERS OF GRAPHITE AND DIAMOND ON SiC FOR CARBON BASED ELECTRONICS

During the course of this research we have studied the dynamics of epitaxial graphene formation on SiC and the production of self standing membranes as well as applications. We believe that the results of these studies will advance the knowledge base for graphene growth. Working with our SBIR partners Structured Materials Industries, Inc, (SMI) the results of our investigations have been used to inform the design and development of a commercial graphene production tool. Such a tool will allow industry to realize a new class of devices with capabilities to transform device performance in multiple areas such as, switching, high frequency analog operation (1), sensing, terahertz (2) generation and terahertz detection.



General Background

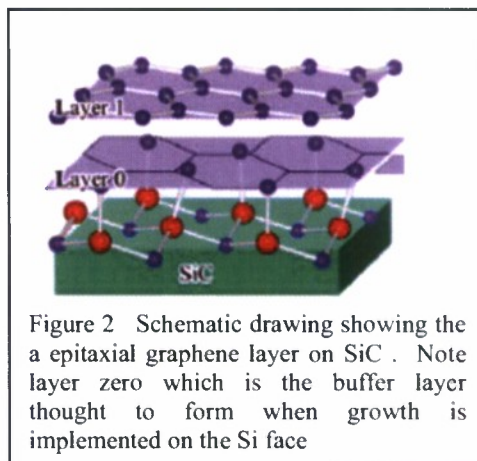
Graphene is a two dimensional single atomic layer of carbon atoms arranged in a honeycomb lattice (3), (4). The Carbon atoms are bonded via sp^2 σ -bonds (see Fig 1). The remaining p-orbitals on each carbon atom stick out of the plane of the carbon atoms and form π -bonds with the p-orbitals of neighboring carbon atoms. The electrons in the π -bonds are delocalized and contribute to carrier transport. The conduction and valence bands resulting from the p-orbitals touch each other at the K and K' points in the first Brillouin zone. Graphene is therefore a zero-bandgap semiconductor. Graphene has a linear energy dispersion relation for both electrons and holes. One of the unique properties of graphene (and of carbon nanotubes) which have generated so much interest in the scientific community is the absence of carrier back-scattering. Back-scattering, where by electrons and holes are scattered from impurities/defects and/or phonons by large angles with respect to their original motion reduces carrier mobility in common semiconductors. In graphene, unlike most other semiconductors, even the periodic part of the electron (hole) Bloch functions are completely orthogonal for electrons (holes) moving in opposite directions. Consequently, impurities/defects and/or phonons cannot easily backscatter carriers. Carrier mobilities in graphene are therefore large and have been measured to be greater than $200,000 \text{ cm}^2/\text{V-sec}$ at low temperatures and are expected to be in the 4-5 million $\text{cm}^2/\text{V-sec}$ range for defect-free graphene at low temperatures (5), (6). These mobility numbers are among the highest of any known semiconductor. The technological importance of graphene is partly due to the large mobilities of both electrons and holes in graphene. If graphene is to compete with silicon for commercial applications, then techniques to reliably and reproducibly produce high quality graphene films on wafer scales are of utmost importance. The research proposed here aims to address the challenges in achieving these goals.

Background epitaxial graphene on SiC

Graphitization of SiC surfaces is a well known phenomenon: shortly after the invention of the Acheson process to produce silicon carbide (then called carborundum) it was observed

that heating of silicon carbide to very high temperatures leads to evaporation of Si and to the formation of graphite (7). The growing interest in SiC as a wide band gap material in the late fifties eventually led to systematic studies of its surfaces. More recent work by I. Forbeaux, et. al. (8) showed in detail the evolution of SiC to graphene. This group showed convincingly that single crystal graphene films can be heteroepitaxially grown on silicon carbide over macroscopic dimensions. However, it was Berger and coworkers (9) (10) (11) (12) who demonstrated the intriguing and potentially useful properties of ultra-thin (several monolayer) graphene layers grown carefully on SiC.

Typical growth conditions for the production of graphene require heating the SiC wafer at temperatures in the range of 1300-1600°C, depending on whether this is performed in ultra high vacuum (UHV) or in Ar at atmospheric pressure. The formation of graphene on the Si face of SiC occurs initially by a sequence of surface reconstructions, the final carbon rich reconstruction serves as the buffer layer for the subsequent growth of the graphene layers. The resulting graphene structure on the Si face of SiC contains a zeroth or buffer layer followed by 1-4 ML as shown in Fig. 2. The exact nature of the zeroth layer is currently a subject of investigation (13). The densities of the C atoms in graphene and in a single SiC bilayer are 32.9 and 10.5 nm⁻², respectively. (8) which requires that the formation of one graphene layer needs the carbon equivalent of about three SiC bilayers. It has been found experimentally that growth of graphene on the Si face is generally self limited to 1-4ML. On the other hand growth on the C face of SiC is dramatically different from growth on the Si face in several ways: 1) the evolution of the surface reconstructions are entirely different 2) the nature of the buffer layer structure (if indeed there is one) is a subject of investigation 3) the growth rate on the carbon face does not appear to be self limited, and 4) the layers grown on the carbon face are rotationally disordered. The rotational disorder on the carbon face has electronic significance (14) and is thought to be responsible for higher mobility on the carbon face as compared to mobility on the Si face. Material on the Si face is bernal stacked and is evolving to the band structure, of graphite in contrast to material on the carbon face, which is electronically equivalent and can be thought of as multi-layers of graphene (14)



Results

Graphene Synthesis

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At Cornell University we have focused on the synthesis of graphene by thermal decomposition of silicon carbide as reviewed in an earlier section. This is the process whereby graphene is formed as epitaxial layers on the surface of silicon carbide wafers as silicon atoms are removed. The process for growth has been shown to be associated with the partial pressure of silicon above the surface during the decomposition process (17). Recognizing that the formation of the graphene layers is dependent on the thermal conditions associated with the release of silicon atoms from the surface and the thermodynamics of the formation of the surface phases during the growth process, we initially utilized a Si containment approach. In this approach, the Si vapors are contained by turning the SiC wafer face downward toward the graphite susceptor. In addition, the SiC wafer is contained within a low profile, covered, oven-like container in the middle of the susceptor which had SiC internal coating that reduced the rate of silicon loss at the growth temperature by providing an additional source of Si atoms in the growth environment. This Si containment technique has allowed us to investigate some of the key conditions for production of high quality graphene. The high temperature growth furnace utilized a small CVD style vacuum chamber as shown in **Fig. 6 (left)**. The growth configuration that allows the study of graphene growth in a downward orientation is also depicted in **Fig. 6.(left)** The close spaced design of the graphite containment region reduces the thermal gradient across the substrate

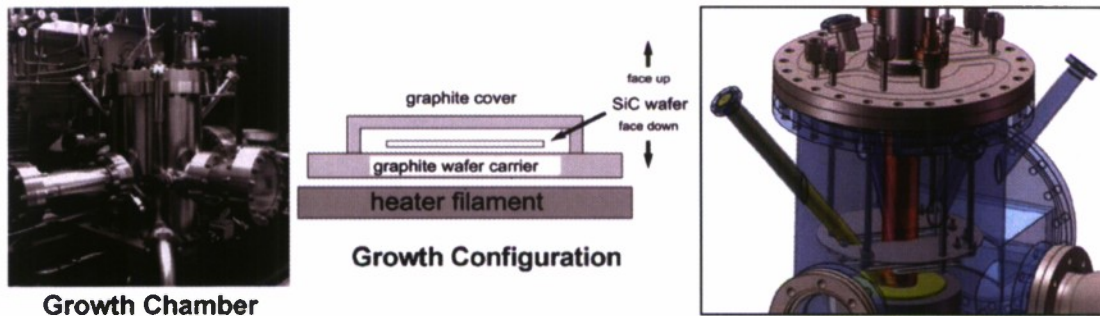
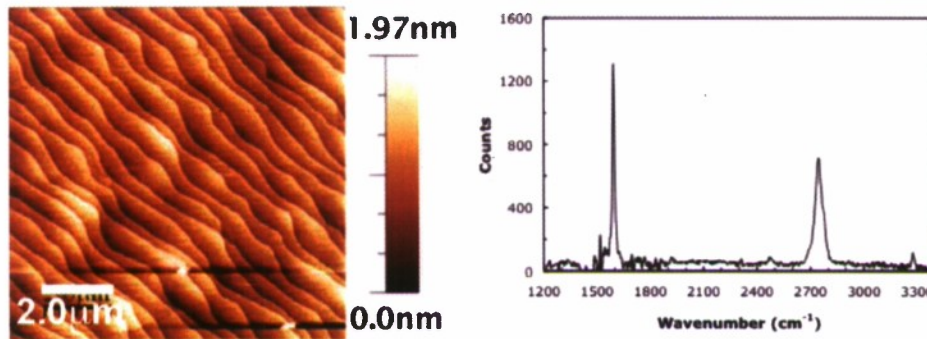


Figure 6. On the left is shown our original graphene growth chamber, shown along with the chamber is a schematic of the wafer carrier or susceptor. The growth orientation for graphene on the SiC faces is depicted. The downward orientation results in a lesser Si flux from the surface and correspondingly thinner layers than the upward direction. On the right is our recently acquired new chamber head with an added upper filament and insitu temperature monitoring capability. The second filament in this chamber accomplishes the same role as the graphene cover shown in the schematic

The SiC coating within the cover inhibits excessive silicon loss from the substrate surface during growth. The lessons learned in our initial experiments have been incorporated into a new reactor design which we have developed in conjunction with SMI Corp **Fig. 6 (right)**. In order to examine the growth characteristics on both the silicon and carbon faces we use semi-insulating SiC with both faces CMP polished. This double sided polishing allowed graphene growth to be conducted on both sides of the wafer with the silicon face-down (carbon face-up) and with the carbon face-down (silicon face-up) simultaneously under the same conditions so that we could examine growth under various levels of silicon flux from the surface. Epitaxial graphene growth was studied in vacuum over a temperature range of 1388°C to 1450°C and variable exposure

times up to 60 minutes. The vacuum pressure varied from the upper 10^{-6} torr range at the lower temperatures to the low 10^{-5} torr range at the highest temperature. Based on the results of our study we have been able to produce high quality graphene on both the Si and C face as indicated by morphological and spectrographic examination. The AFM image in **Fig. 7** shows



morphology achieved on the Si face. A standard method for

Figure 7 a) AFM of the morphology of graphene grown on the Si face of SiC b) The Raman spectra are shown a very low disorder ratio, I_D/I_G , is indicated by the very small D peak near 1360cm^{-1}

characterizing the disorder in graphene films is Raman. (21) (22) This disorder is characterized by the disorder ratio, I_D/I_G , determined as the ratio of the peak heights for two prominent peaks in the Raman spectrum (**Fig. 8**) for graphene; the D peak near 1370 cm^{-1} ; and the G peak near 1580 cm^{-1} . (23) (24) (25) Raman results indicated disorder characteristics, represented by the intensity ratio, I_D/I_G , down to a very low value <0.02 . Folds have been observed to occur predominately on the carbon face. The SEM images in **Fig 8** show a cross section on one of our

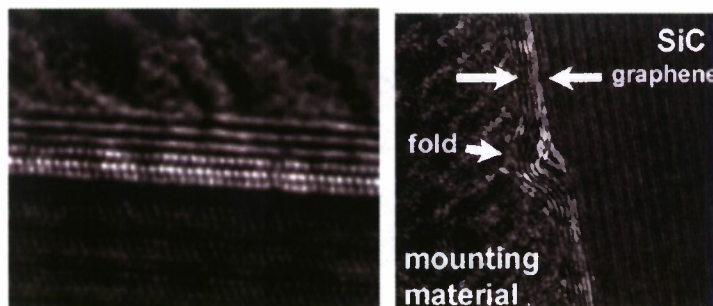


Figure 8 TEM micrograph showing the cross section of one of our graphene layers grown on (0001) SiC (left). TEM micrograph of a fold (right)

Si face samples (left) and a cross section of a fold (right). Since folds occur much more extensively on the carbon face, a goal of our work has been the successful reduction of the number and extent of these features. Folds are expected to affect the electrical transport characteristics and the ability to fabricate structures on the graphene layers. The AFM images in **Fig 9** indicate the degree of reduction in fold structure and

occurrence of pits that has been achieved as a result of our graphene synthesis studies. The reduction in fold size and extent appears to correlate with a reduction in the thickness of the graphene layer on the carbon face. The AFM images in **Fig. 9** show that the underlying step structure in the SiC substrate can now be observed on our C face samples since the relative thinness of the graphene layers can now conform with the SiC surface. Most of the improvement has been achieved by dynamically varying the growth characteristics to adjust the rate of nucleation and graphene layer formation and by minimizing the variation in surface stress during

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the cool down stage after growth. The results of our efforts have determined that folds can be minimized.

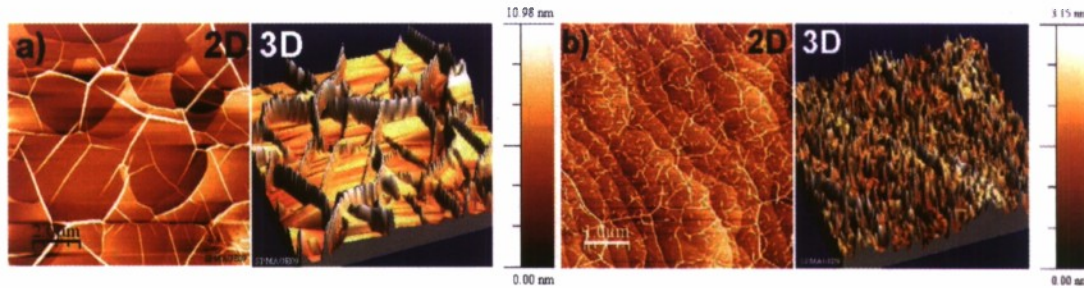


Figure 9. AFM images show the improvement in C face graphene achieved during our study. Image (a) shows the extent of the folds in a relatively thick graphene layer on the carbon face estimated to be about 25 to 30 monolayers. Image (b) is carbon face graphene that's about 5 to 8 monolayers thick.

Graphene Membranes

Graphene membranes were formed by patterning and subsequently etching full surface graphene formed as described above. SiC is chemically a highly resistant material and it is typically patterned using dry etching techniques. However, the plasmas involved in the dry etching process will destroy the graphene. Hence, a wet etching process was developed to etch

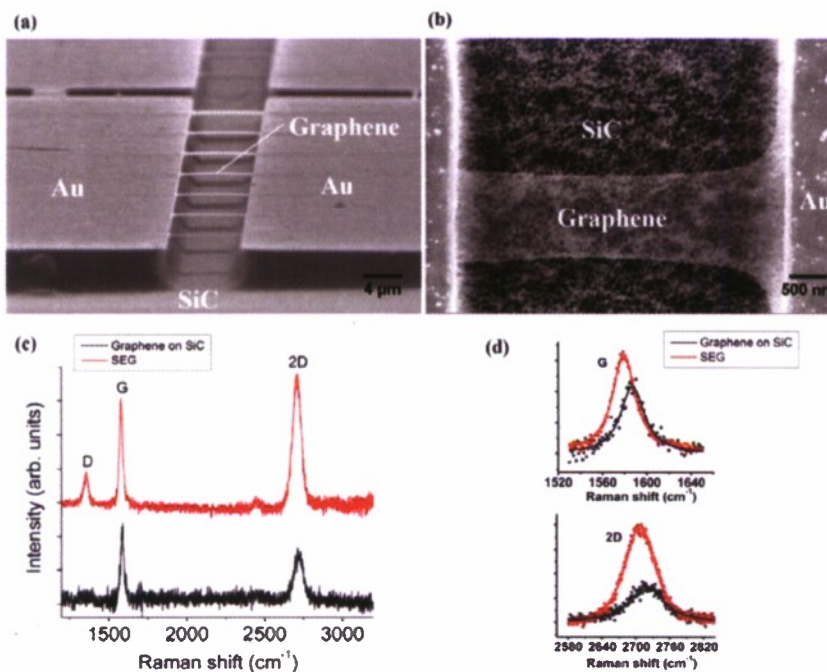


Figure 10 a-b) Electron micrographs of freestanding membranes of graphene c) Raman spectra of graphene before (black) and after (red) release from substrate. d) Blue shift of G and 2D Raman peaks (black before release) (red after release)

the SiC and suspend graphene.. A scanning electron microscope (SEM) image of arrays of doubly-clamped SEG devices produced by this technique is shown in **Fig. 10(a)**. This technique works even for extremely thin (few layer) graphene, as evidenced by the electron transparency of the device shown in **Fig. 10(b)**. Devices with dimensions ranging from 3 – 20 μm in length and 0.5 – 3.5 μm in width were successfully produced. The thickness of the graphene devices used in this study is estimated to be 1 nm graphene, as evidenced by the electron transparency of the device shown in **Fig. 10(b)**. Raman

spectroscopy was performed on the graphene before and after the photoelectrochemical etch process. Raman spectra were collected using a Renishaw InVia micro-Raman system with an excitation wavelength of 488 nm. A blue-shifted G-peak at $\sim 1587 \text{ cm}^{-1}$ and a 2D peak at $\sim 2719 \text{ cm}^{-1}$ are observed for graphene on SiC (after background subtraction) as shown in Fig 3(a). This blue-shift has been attributed to compressive strain in the graphene grown over SiC (26). A disorder related D-peak is barely visible. The Raman spectrum of a SEG device is shown in Fig. 10(b). The G peak is seen at $\sim 1580 \text{ cm}^{-1}$ and the 2D peak is at $\sim 2707 \text{ cm}^{-1}$. These red-shifts from the graphene-on-SiC peaks agree with the hypothesis that registry with the substrate is the cause of the original blue-shift.

Highly Sensitive and Selective NO₂ Sensing using Epitaxial Graphene on 6H-SiC

Using Epitaxial graphene grown on SiC substrate described above we have investigated gas sensing. Our experimental results demonstrate graphene layers grown on both Si and C-faces of semi-insulating 6H-SiC can offer very high detection sensitivity, selectivity, and fast response time. Exposure to only 500 ppb NO₂ reduced the conductivity by 2.25%, while 18 ppm caused a reduction of $\sim 10\%$ (see Fig 11). In contrast, high concentration of commonly interfering gases, namely, CO₂ (20%), H₂O (saturated vapor), NH₃ (550 ppm), and pure O₂ increased the conductivity by a maximum of only $\sim 2\%$. Graphene on C-face of SiC resulted in somewhat lower sensitivity for the test gases, with the conductivity changing in opposite direction compared to Si-face for any particular gas. The conductance change due to molecular adsorption was compared to changes in surface work function (SWF) as well as the charge transfer between the adsorbed molecules and the graphene surface. Measurements conducted at higher

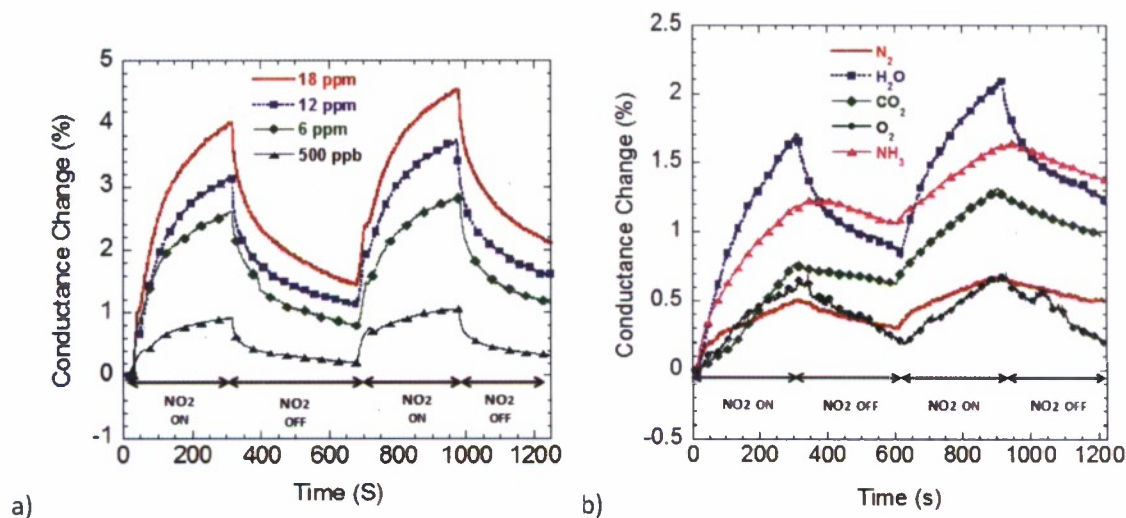


Figure 11: a) The change of conductance of graphene formed on the C-face of SiC for various amounts of NO₂.
 b) The change of conductance in epitaxial graphene layers grown on (a) Si- face, and (b) C-face of SiC for different concentrations of N₂, NH₃, CO₂, O₂, and H₂O

temperature showed significantly larger change in conductivity and faster response time.

Publications resulting from this award-Publications from this work have dealt with the growth studies discussed above (27) (28), as well as the fabrication of free standing membranes of graphene (discussed above) (29) and characterization studies (30) (31) (32) to develop device quality material for analog and terahertz applications

Relationship with SBIR partner

As part of a related effort we are in a collaborative effort with SMI for the development of a commercial reactor for the production of graphene. SMI and Cornell University are developing a flexible graphene film deposition system, for both research and production applications. The proposed graphene tool will accommodate wafer sizes up to 6 inch, and will be scalable to larger wafers or multiple (large) wafers for high volume production. The

graphene film production tool (shown in Fig. 12) is based on thermal decomposition of silicon carbide (SiC). The tool will be high vacuum compatible, with provisions for a wide range of gas inputs, enabling gas phase doping of graphene, or combining the graphene process with other techniques such as chemical vapor deposition (CVD). The proposed tool will also have full provisions for in-situ monitoring during processing; including: optical and pyrometry, among others. The overall tool is designed around processes demonstrated at Cornell and elsewhere and SMI's multiple commercial reactor tool technologies. Since the recent discovery of graphene has sparked intense research, a critical need exists for reliable and reproducible production tools for graphene films. The tool platform should be scalable from initial research size samples to ultimate high volume production, for smooth transition of technical developments to the marketplace. SMI is an established developer and supplier of thin film deposition tools for advanced materials, ranging from ferroelectrics to electro-optic materials to wide bandgap semiconductors. In Phase I, SMI and Cornell facilities at Cornell University have been used to refine and enhance the process window and tooling already established at Cornell for graphene film production. SMI will design an alpha prototype of a graphene production tool, to be built in Phase II, which can operate over the entire process window.

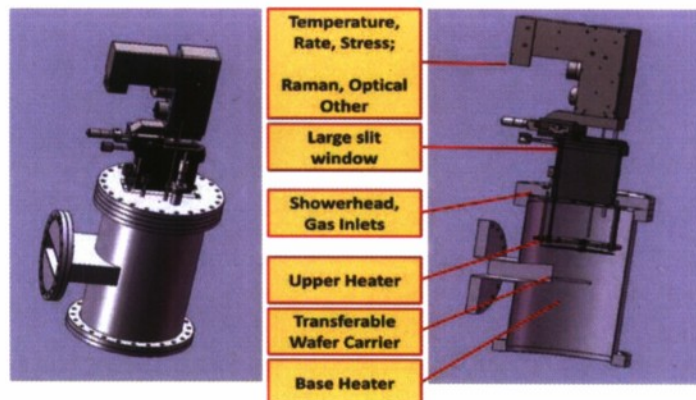


Figure 12 Schematic diagram of proposed production graphene reactor based upon existing Cornell reactor.

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